

1. FM PTG-1350  
(REV. 7-99)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

3229-4003

**TRANSMITTAL LETTER TO THE UNITED STATES  
DESIGNATED/ELECTED OFFICE (DO/EO/US)  
CONCERNING A FILING UNDER 35 U.S.C. 371**

U.S. APPLICATION NO. (If known, see 37 CFR 1.5)

**09/581885**INTERNATIONAL APPLICATION NO.  
PCT/FI98/00993INTERNATIONAL FILING DATE  
18 December 1998 (18.12.98)PRIORITY DATE CLAIMED  
19 December 1997 (19.12.97)

TITLE OF INVENTION BLEACHING OF CHEMICAL PULP WITH PERACID

APPLICANT(S) FOR DO/EO/US

Jukka JÄKÄRÄ, Juha PATOLA, and Aarto PAREN

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
  - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
  - b. ☒ has been transmitted by the International Bureau.
  - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
  - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
  - b. ☐ have been transmitted by the International Bureau.
  - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
  - d. ☐ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment.  
☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
14. ☒ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☒ Other items or information: Copy of first page of published international application number PCT/FI98/00993 (publication number WO 99/32710)

097581885

17. ☐ The following fees are submitted:**BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)):**

Neither international preliminary examination fee (37 CFR 1.482)  
nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO  
and International Search Report not prepared by the EPO or JPO ..... \$970.00

International preliminary examination fee (37 CFR 1.482) not paid to  
USPTO but International Search Report prepared by the EPO or JPO ..... \$840.00

International preliminary examination fee (37 CFR 1.482) not paid to USPTO but  
international search fee (37 CFR 1.445(a)(2)) paid to USPTO ..... \$690.00

International preliminary examination fee paid to USPTO (37 CFR 1.482)  
but all claims did not satisfy provisions of PCT Article 33(1)-(4) ..... \$670.00

International preliminary examination fee paid to USPTO (37 CFR 1.482)  
and all claims satisfied provisions of PCT Article 33(1)-(4) ..... \$96.00

**ENTER APPROPRIATE BASIC FEE AMOUNT =****CALCULATIONS** PTO USE ONLY

\$ 840.00

Surcharge of \$130.00 for furnishing the oath or declaration later than ☐ 20 ☐ 30  
months from the earliest claimed priority date (37 CFR 1.492(e)).

\$ --

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE
Total claims	16 - 20 =	--	X \$18.00
Independent claims	2 - 3 =	--	X \$78.00
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+ \$260.00

\$ --

\$ --

\$ --

**TOTAL OF ABOVE CALCULATIONS =**

\$

Reduction of 1/2 for filing by small entity, if applicable. A Small Entity Statement  
must also be filed (Note 37 CFR 1.9, 1.27, 1.28).

\$

**SUBTOTAL =**

\$ 840.00

Processing fee of \$130.00 for furnishing the English translation later than ☐ 20 ☐ 30  
months from the earliest claimed priority date (37 CFR 1.492(f)).

\$

+

**TOTAL NATIONAL FEE =**

\$ 840.00

Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be  
accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property

\$

+

**TOTAL FEES ENCLOSED =**

\$

Amount to be  
refunded:

charged:

\$ 840.00

a. ☐ A check in the amount of \$\_\_\_\_\_ to cover the above fees is enclosed.

b. ☒ Please charge my Deposit Account No. 13-4500 in the amount of \$ 840.00 to cover the above fees.  
A duplicate copy of this sheet is enclosed. (Order No. 3229-4003)

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any  
overpayment to Deposit Account No. 13-4500. A duplicate copy of this sheet is enclosed.  
(Order No. 3229-4003)

**NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.**

SEND ALL CORRESPONDENCE TO:

Israel Blum  
Morgan & Finnegan, L.L.P.  
345 Park Avenue  
New York, New York 10154  
Tel. No. (212) 758-4800  
Fax No. (212) 751-6849

SIGNATURE:

Israel Blum

NAME

26,710

REGISTRATION NUMBER

**430 Rec'd PCT/PTO 19 JUN 2000**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE (DO/EO/US)

Applicant(s) : Jukka JÄKÄRÄ, et al.  
International  
Application No. : PCT/FI98/00993  
International  
Filing Date : December 18, 1998  
U.S. Serial No. : To be assigned  
U.S. Filing Date : June 19, 2000  
Group Art Unit : To be assigned  
Examiner : To be assigned  
For : **BLEACHING OF CHEMICAL PULP  
WITH PERACID**

PRELIMINARY AMENDMENT

Assistant Director of Patents  
Box PCT  
Washington, D.C. 20231

Attention: DO/EO/US

S I R:

Please amend the above-identified patent application, filed of even date herewith for the specific purpose of eliminating multiple dependent claims and multiple dependent claims fees.

In the Claims:

1. (Amended) [A] In a method for the bleaching of chemical pulp, wherein the pulp is treated in a plurality of different steps and wherein at least in one step a bleaching solution which contains a peracid is used, [characterized in

that] the improvement in which the peracid is used in an post-bleaching which is the last step of the bleaching process, the post-bleaching taking place in the presence of one or several earth-alkali metal compounds.

2. (Amended) A method according to Claim 1, [characterized in that] wherein the brightness of the pulp before the post-bleaching carried out with a peracid is at a minimum 85% ISO.

3. (Amended) A method according to Claim 1 [or 2, characterized in that] wherein the kappa number of the pulp before the post-bleaching with a peracid is at a maximum 4.

4. (Amended) A method according to [any of the above claims, characterized in that] claim 1, wherein the amount of the peracid used for the post-bleaching is 0.1-7 kg/tp[, preferably 0.5-3 kg/tp].

5. (Amended) A method according to claim 4, [characterized in that] wherein the peracid is peracetic acid.

6. (Amended) A method according to [any of the above claims, characterized in that] claim 1, wherein the post-bleaching solution contains a calcium compound[, such as calcium acetate or calcium carbonate].

7. (Amended) A method according to [any of the above claims, characterized in that] claim 1, wherein the post-bleaching solution contains a magnesium compound[, such as magnesium sulfate].

8. (Amended) A method according to [any of the above claims, characterized in that] claim 1, wherein the pH of the

post-bleaching solution is within the range of 3-8[, preferably 4-7].

9. (Amended) A method according to [any of the above claims, characterized in that] claim 1, wherein the post-bleaching is carried out after the bleach plant steps in a pulp flow pipe, a storage tower and/or the paper machine.

10. (Amended) A method according to [any of the above claims, characterized in that] claim 1, wherein the post-bleaching is carried out in the paper machine or elsewhere at a paper mill.

11. (Amended) The use of a solution which contains a peracid [or] and an earth-alkali metal for the post-bleaching of a delignified pulp at a paper mill.

Please add new claims 12-16:

12. (New) A method according to claim 2 wherein the kappa number of the pulp before the post-bleaching with a peracid is at a maximum 4.

13. (New) A method according to claim 4, wherein the amount of the peracid used for the post-bleaching is 0.5-3 kg/hp.

14. (New) A method according to claim 6, wherein the calcium compound is calcium acetate or calcium carbonate.

15. (New) A method according to claim 7, wherein the magnesium compound is magnesium sulfate.

16. (New) A method according to claim 8, wherein the pH is within the range of 4-7.

R E M A R K S

The amendments to the claims do not present new matter. Claims 1-11 have been amended (1) to eliminate multiple dependency among the claims and thus avoid the need to pay the multiple dependency fees on filing this application; and (2) to correct informalities in the claims. In addition new claims 12-16 have been added to claim subject matter recited in original claims 2, 4, 6, 7 and 8, respectively, which subject matter was deleted from amended claims 2, 4, 6, 7 and 8 to avoid informality objections.

Please charge Deposit Account No. 13-4500, Order No. 3229-4003 for any additional fees, which may be required or credit any overpayment to that account. A duplicate copy of this sheet is enclosed.

Respectfully submitted,

*Israel Blum*

---

Israel Blum  
Registration No. 26,710

MORGAN & FINNEGAN, L.L.P.  
345 Park Avenue  
New York, New York 10154  
Tel. No. (212) 758-4800  
Fax No. (212) 751-6849

BLEACHING OF CHEMICAL PULP WITH PERACID

5 The present invention relates to a method for the bleaching of  
chemical pulp, wherein the pulp is treated in a plurality of dif-  
ferent steps and a solution containing a peracid is used in at  
least one of the steps. The invention additionally relates to the  
10 use of a peracid-containing bleaching solution in a process step  
according to the invention.

004150-522350  
15 In a prior known pulp delignification process, a raw pulp ob-  
tained by digesting cellulose-containing materials with suitable  
digestion chemicals is subjected to a treatment wherein the  
lignin is removed and a bleaching which involves the use of  
oxidative chemicals is carried out. The purpose of conventional  
bleaching of chemical pulp is to bring to completion the removal  
of lignin from a raw pulp obtained from a digestion procedure.  
This bleaching can be carried out by using chlorine or chlorine  
20 dioxide and thereafter alkaline extraction steps in which the  
lignin is dissolved out of the pulp. At present, the bleaching of  
a chemical pulp is increasingly often carried out by bleaching  
methods using no elementary chlorine or chlorine compounds. The  
first-mentioned bleaching is called ECF (elementary chlorine  
25 free) bleaching and the latter TCF (totally chlorine free)  
bleaching. The chemical pulp is bleached in several successive  
steps. Nowadays the bleaching is often started with an oxygen  
delignification, whereafter further bleaching can be carried out  
by various methods. In TCF bleaching, delignification can be  
30 continued, for example, by using ozone, peracetic acid or  
hydrogen peroxide in acid or alkaline conditions. In ECF  
bleaching, chlorine dioxide steps and thereafter alkaline  
extraction steps are used.

35 There is a previously known method in which the last bleaching  
step for chemical pulp is carried out by using peracetic acid.

Peracetic acid can be prepared most simply by mixing vinegar and hydrogen peroxide, whereupon a so-called equilibrium solution is obtained which contains, in addition to peracetic acid, also unreacted initial substances. Also a pure peracetic acid solution  
5 can be prepared from this reaction solution by distillation. There are also known other methods for preparing peracetic acid.

Peracetic acid (PAA) is a highly selective bleaching chemical by the use of which good strength properties of the pulp can be  
10 maintained. When peracetic acid is used, the optimum pH is approx. 4-7. At a lower pH peracetic acid is a highly selective delignifying chemical. At a higher pH its bleaching action is greater but its selectivity decreases.

By modern bleaching methods, high degrees of brightness can easily be achieved with both ECF and TCF pulps. However, achieving very high degrees of brightness by conventional methods may consume considerable amounts of chemicals. Likewise, after a normal bleaching the variation of the degree of brightness of the  
15 pulp and the pH of the stock going to the paper machine may cause runnability problems in the paper machine. Pulp brightness may also be reduced in storage towers, in which case the pulp must be bleached clearly beyond the required degree in order for the reduction of brightness to be compensated for.  
20

A good method for solving these problems is post-bleaching. Agents suitable for post-bleaching include peracetic acid and dithionite. The use of hydrogen peroxide is limited by the fact that in order to function properly it would require alkaline  
25 conditions. With respect to pH, dithionite and peracetic acid are highly suited for post-bleaching. Dithionite is used commonly in the bleaching of mechanical pulps, but normally it is not used in the bleaching of chemical pulp. The sulfur present in it may also cause problems. Post-bleaching with peracetic acid is a very  
30 effective method of increasing brightness. The consumption of chemicals for bleached pulps is low, and in addition the reaction  
35



is rapid even at low consistency. Furthermore, the optimum pH range for bleaching is precisely suitable for post-bleaching. However, the peracetic acid post-bleaching of chemical pulps involves the problem that, within the pH range optimal for the bleaching result, selectivity is no longer at its best. In the case of a completely bleached pulp having a very low kappa number, the use of peracetic acid damages the fibers, causing the breaking down of carbohydrates and the lowering of pulp strength. This also causes dissolving of organic matter out of the pulp, a factor which may disturb the running of the paper machine.

The bleaching method according to the invention, which solves the problems mentioned above, is characterized in that a peracid is used in the post-bleaching which is the last step of the bleaching process and takes place in the presence of one or more earth-alkali metal compounds. An post-bleaching such as this can advantageously be carried out as a separate step at the paper mill to which the pulp has been transferred from a bleach plant.

The earth-alkali metals, such as magnesium and calcium, used simultaneously with a peracid in the method according to the invention stabilize carbohydrates so that the reducing of the pulp strength is prevented. In other words, the strength of the pulp remains good and the dissolving of organic matter is insignificant. The brightness and whiteness of the pulp are also higher than without the use of earth-alkali metals. This decreases, for example, the need for using optical brighteners in the paper machine. When this method is used, the pH of the stock to be turned into paper, and the brightness and whiteness of the pulp are more even, in which case the runnability of the paper machine is considerably improved.

The adding of magnesium to the pulp, for example in oxygen- and peroxide-based steps, is prior art. Likewise, the use of magnesium in connection with peracetic acid delignification is known from, for example, the publication Liebergott, N., 81st annual

meeting, technical section CPPA (1995), B 157 - B 170. According to the publication, magnesium protects the viscosity of the pulp. The question is, however, of delignification which is carried out on a pulp having a high kappa number. This delignification step is followed by a separate bleaching during which the brightness is raised to its final level. During the delignification, transition metals such as Fe and Mn are released from the pulp, and magnesium prevents their detrimental effects. The addition of magnesium is used for improving the ratio of magnesium to transition metal, which improves especially the running of the subsequent alkaline peroxide step.

In contrast to the foregoing, the invention concerns post-bleaching which is carried out on an already delignified pulp having a low kappa number (preferably below 4) and being free of transition metals, the purpose being to raise the brightness and whiteness of the pulp to the desired level. In this case the peracetic acid turns colorless the chromophoric groups in the pulp, mainly in carbohydrates. In the above-mentioned state-of-the-art methods the question is of the reactions of peracetic acid with residual lignin in the pulp.

Another difference between the prior-art peracetic acid delignification method and the present invention is that in the invention the peracetic acid dose is considerably smaller. In the method according to the invention, surprisingly even a very small peracetic acid dose, 0.1-7 kg/tp, preferably 0.5-3 kg/tp, is sufficient, larger doses being even detrimental.

In methods using peracetic acid known from the literature, it has been observed that the optimum pH with respect to brightness is within a neutral or mildly alkaline range; the strength of the pulp suffers within a slightly acid range. In the method according to the invention it is possible to operate within a pH range of 3-8, preferably 4-7, and at the same the decreasing of the pulp strength is prevented. A preferably acid pH range is

essential for the runnability of the paper machine. It has been observed that by using an earth-alkali metal addition it is possible to carry out post-bleaching within an acid pH range without a loss of pulp strength and without organic matter being dissolved out of the pulp. In prior known methods the best bleaching effect was achieved in mildly alkaline conditions. Since the stability of peracetic acid decreases as the pH rises, when small peracetic acid doses are used wasteful consumption of the chemical decreases brightness. With large peracetic acid doses the effect of wasteful consumption of the chemical does not show. Thus by the method according to the invention it is possible to after-bleach pulp with considerably smaller chemical doses than by prior known methods, without a loss of pulp strength.

Another advantage gained through an post-bleaching carried out using a peracid, e.g. peracetic acid, within an acid pH range is that the acid released during the reaction serves as an acidification chemical, whereupon the use of  $\text{SO}_2$  or  $\text{H}_2\text{SO}_4$  as an acidification chemical is avoided. Thus the total sulfur amount can be lowered and possible  $\text{SO}_2$  odor problems are avoided.

The peracid used in the method is preferably peracetic acid. Other usable peracids include performic acid, perpropionic acid or some longer-chain percarboxylic acid. The peracid may be a so-called equilibrium solution, i.e. a reaction mixture containing the said acid, a peracid and hydrogen peroxide, or a pure peracid solution can be used. The method by which the peracid solution is prepared does not restrict the use of the method; the peracid may be prepared, for example, by distilling an equilibrium solution or from an anhydride and hydrogen peroxide. The peracid may also be a monopersulfuric acid (Caro's acid) or a mixture of Caro's acid and a percarboxylic acid, e.g. peracetic acid. Persulfates may also be used, either as such or as a mixture with any of the above-mentioned peracids.

The earth-alkali metal used in the post-bleaching according to the invention may be in particular calcium or magnesium. These may be added to the bleaching solution in the form of sulfate, acetate, carbonate, oxide or any other compound. It is possible to use in the method either magnesium or calcium alone or both of them together at any ratio. Calcium has over magnesium the advantage that calcium will not precipitate extract-based and resin-based substances which may be present at the wet end of the paper machine.

The post-bleaching according to the invention may be preceded by any known bleaching reaction known from bleaching sequences. The post-bleaching is especially advantageous for use after chlorine dioxide or peroxide bleaching. Also, acidification possibly taking place after the bleaching does not restrict the application of the method; it can be used on both non-acidified and acidified stock. The said acidification may be carried out with, for example,  $\text{SO}_2$ , sulfuric acid or any acid suitable for this purpose. The functioning of the invention is not affected by a possible wash of the pulp before or after the post-bleaching, or by the absence of a wash. The consistency of the pulp to be after-bleached may be 1-30 % and the temperature during the post-bleaching may be between 30 and 100 °C.

The after-bleached pulp may be taken directly to the paper machine or be dried to produce baled pulp.

The method is suited for use on sulfate and sulfite pulps made both from softwood and from hardwood, and on various organosolv pulps.

The said prior art methods in which a peracid and magnesium are used or in which a peracid has been present in the last steps of the bleaching sequence are characterized in that they are carried out in the normal manner in the bleach plant of the pulp mill. The post-bleaching according to the invention, in which the

bleaching reaction is very rapid even at a low consistency, is not tied to the bleach plant and thus also does not require investment in equipment for the bleach plant. As was already pointed out, the invention primarily concerns an post-bleaching carried out after the bleaching sequence of the bleach plant, outside the actual bleaching, for example in the pulp flow pipe during the transfer of pulp, during pulp storage or in the paper machine. Instead of the bleach plant, the place at which the post-bleaching according to the invention is carried out is typically a storage tower for bleached pulp or the paper mill.

The earth-alkali metal compounds used can be added to the bleaching solution in any suitable process step. They can be added to the circulating waters before the water arrives in the post-bleaching step, or they may, for example, come in the dilution waters from the paper machine, where they may be added in any form. It is also possible to use chelators simultaneously with earth-alkali metals.

In addition to the post-bleaching method described above, the invention comprises the use of a solution which contains a peracid and an earth-alkali metal for post-bleaching of delignified pulp at a paper mill.

#### Example 1

A birch sulfate pulp which had been bleached using a sequence consisting of an oxygen step, chelation, an oxygen + peroxide step, a chlorine dioxide step, a peroxide extraction, a chlorine dioxide step (-O-Q-Op-D-Ep-D) was subjected separately to an post-bleaching at 50 °C with a 30 min retention at a consistency of 5 % with a peracetic acid dose of 3 kg/tp at two different pH levels (approx. 6.5 and approx. 4.5). The peracetic acid used had been distilled. Calcium was added to the pulp in the form of acetate, and magnesium in the form of sulfate. The figures in the table indicate the dose of the chemical concerned per one metric

ton of pulp (kg of 100 % chemical/one metric ton of pulp). Experiment number 0 stands for pulp which has not been after-bleached.

5							
	Exp.	Initial	Final	CaOAc	MgSO <sub>4</sub>	Brightness	Viscosity
	No.	pH	pH	kg/tp	kg/tp	% ISO	dm <sup>3</sup> /kg
	0	-	-	-		90.3	930
10	1	6.5	5.4	-		91.0	766
	2	4.5	4.3	-	-	91.5	845
15	3	6.5	5.3	-	1	92.5	925
	4	4.5	4.2	-	1	92.3	911
	5	6.5	5.4	1	-	92.8	899
20	6	4.5	4.3	1	-	92.6	901
	7	6.5	5.3	0.5	0.5	92.8	887
25	8	4.5	4.4	0.5	0.5	92.5	896

As can be seen from the table, with the earth-alkali addition the viscosity of the pulp can be maintained clearly higher. The viscosity determined by the copper-ethylene diamine method is directly proportional to the strength of the pulp, especially when the same pulp sample is concerned. It is well-founded to assume that the higher the viscosity of the pulp, the better its strength. A higher viscosity also means that the yield is better and that less organic matter has been dissolved out of the pulp.

ISO brightness was improved by approx. 1 % ISO by the earth-alkali addition.

### Example 2

5

A birch sulfate pulp which had been bleached by the sequence -O-Q-Op-D-Ep-D according to Example 1 was subjected to an post-bleaching at 50 °C with a 30 min retention at a consistency of 5 % with a peracetic acid dose of 3 kg/tp at different pH levels. The magnesium sulfite addition was 1 kg/tp. The peracetic acid used had been distilled. The brightness of the pulp before the post-bleaching was 90,3 % ISO and viscosity 930 dm<sup>3</sup>/kg.

10

15

20

25

30

35

No Mg addition			MgSO <sub>4</sub>		
Initial	Viscosity	Brightness	Viscosity	Brightness	
pH	dm <sup>3</sup> /kg	% ISO	dm <sup>3</sup> /kg	% ISO	
3	890	91.4	913	92.0	
3.5	868	91.4	912	92.1	
4	850	91.5	907	92.2	
4.5	839	91.5	911	92.3	
5	799	91.6	912	92.4	
5.5	766	91.7	915	92.5	
6.5	750	91.8	925	92.5	
8	802	91.2	916	91.9	

It can be seen from the table that without a magnesium addition the viscosity of the pulp is lowered considerably; at its lowest the viscosity is within a pH range of approx. 5.5-6. Owing to the magnesium addition the viscosity remains high regardless of the pH. Brightness is also clearly higher.

### Example 3

A TCF-bleached softwood sulfate pulp which had been bleached using oxygen, ozone and hydrogen peroxide was after-bleached at 70 °C with a 240 min retention at a consistency of 10 % by using two different peracetic acid doses. The conditions and results of the post-bleaching are shown in Table 3. The brightness of the pulp before the post-bleaching was 86.8 % ISO and viscosity 642 dm<sup>3</sup>/kg, and the kappa number was 1.7.

Experiment No.	1	2	3	4
PAA, kg/tp	3	1.5	1.5	3
pH, initial	7.2	7.2	7.2	7.2
pH, final	6.3	6.6	6.8	5.6
MgSO <sub>4</sub> , kg/tp	1	1	-	-
Residual PAA, kg/tp	0.7	0.5	0.1	0.1
Kappa	1.6	1.6	1.4	1.3
Viscosity, dm <sup>3</sup> /kg	633	625	572	564
Brightness, % ISO	89.1	88.9	88.4	88.6



As is seen from the table, with the magnesium addition a higher brightness was obtained and the viscosity remained clearly better. In post-bleaching with a magnesium addition, no organic matter was dissolved, which can be seen from the kappa number and

5 viscosity of the pulp.

#### Example 4

A TCF-bleached softwood sulfate pulp which had been bleached using oxygen, ozone and hydrogen peroxide was after-bleached at 70 °C with a 240 min retention at a viscosity of 10 % by using two different peracetic acid doses. The conditions and results of the post-bleaching are shown in Table 4. The brightness of the pulp before the post-bleaching was 87.6 % ISO and viscosity

15 623 dm<sup>3</sup>/kg, and the kappa number was 1.8.

Experiment No.	1	2	3	4	5	6
PAA, kg/tp	1.5	1.5	1.5	1.5	1.5	1.5
pH, initial	7.2	5.5	5.5	6.1	6.5	7.2
pH, final	6.9	4.9	4.9	5	5.5	6.9
MgSO <sub>4</sub> , kg/tp	1	1	-	-	-	-
Residual PAA, kg/tp	0.4	0.4	0.4	0.4	0.4	0.2
Viscosity, dm <sup>3</sup> /kg	612	609	574	571	561	579
Brightness, % ISO	89.9	89.3	88.6	88.5	88.5	89.0

As can be seen from the table, a clearly better final viscosity is obtained with the magnesium addition.

For a person skilled in the art it is clear that the various applications of the invention are not limited to those presented above by way of example only; they may vary within the  
5 accompanying patent claims.

## Claims

1. A method for the bleaching of chemical pulp, wherein the pulp is treated in a plurality of different steps and wherein at least in one step a bleaching solution which contains a peracid is used, **characterized** in that the peracid is used in an post-bleaching which is the last step of the bleaching process, the post-bleaching taking place in the presence of one or several earth-alkali metal compounds.

2. A method according to Claim 1, **characterized** in that the brightness of the pulp before the post-bleaching carried out with a peracid is at minimum 85 % ISO.

3. A method according to Claim 1 or 2, **characterized** in that the kappa number of the pulp before the post-bleaching with a peracid is at maximum 4.

4. A method according to any of the above claims, **characterized** in that the amount of the peracid used for the post-bleaching is 0.1-7 kg/tp, preferably 0.5-3 kg/tp.

5. A method according to Claim 4, **characterized** in that the peracid is peracetic acid.

6. A method according to any of the above claims, **characterized** in that the post-bleaching solution contains a calcium compound, such as calcium acetate or calcium carbonate.

7. A method according to any of the above claims, **characterized** in that the post-bleaching solution contains a magnesium compound, such as magnesium sulfate.

8. A method according to any of the above claims, **characterized** in that the pH of the post-bleaching solution is within the range 3-8, preferably 4-7.

9. A method according to any of the above claims, **characterized** in that the post-bleaching is carried out after the bleach plant steps in a pulp flow pipe, a storage tower and/or the paper machine.

10. A method according to any of the above claims, **characterized** in that the post-bleaching is carried out in the paper machine or elsewhere at a paper mill.

11. The use of a solution which contains a peracid or an earth-alkali metal for the post-bleaching of a delignified pulp at a paper mill.

004150 "SECRET" 004150

to Rec'd PCT/PTO 14 SEP 2000

Docket No. 3229-4003

**COMBINED DECLARATION AND POWER OF ATTORNEY FOR  
ORIGINAL, DESIGN, NATIONAL STAGE OF PCT, SUPPLEMENTAL  
DIVISIONAL, CONTINUATION OR CONTINUATION-IN-PART APPLICATION**

As a below name inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

BLEACHING OF CHEMICAL PULP WITH PERACID  
the specification of which

- a. ☐ is attached hereto
- b. ☒ was filed on June 19, 2000 as application Serial No. 09/581,885 and was amended on June 19, 2000 (if applicable).

**PCT FILED APPLICATION ENTERING NATIONAL STAGE**

- c. ☒ was described and claimed in International Application No. PCT/FI98/00993 filed on December 18, 1998 and as amended on \_\_\_\_\_ (if any).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the patentability as defined in Title 37, Code of Federal Regulations, § 1.56.

I hereby specify the following as the correspondence address to which all communications about this application are to be directed:

SEND CORRESPONDENCE TO: MORGAN & FINNEGAN, L.L.P.  
345 Park Avenue  
New York, N.Y. 10154

DIRECT TELEPHONE CALLS TO: ISRAEL BLUM  
(212) 758-4800

☐ I hereby claim foreign priority benefits under Title 35, United States Code § 119(a)-(d) or under § 365(b) of any foreign application(s) for patent or inventor's certificate or under § 365(a) of any PCT international application(s) designating at least one country other than the U.S. listed below and also have identified below such foreign application(s) for patent or inventor's certificate or such PCT international application(s) filed by me on the same subject matter having a filing date within twelve (12) months before that of the application on which priority is claimed:

☒ The attached 35 U.S.C. § 119 claim for priority for the application(s) listed below forms a part of this declaration.

004760-504050



Hughes (Reg. No. 26,914), William S. Feiler (Reg. No. 26,728), Joseph A. Calvaruso (Reg. No. 28,287), James W. Gould (Reg. No. 28,859), Richard C. Komson (Reg. No. 27,913), Israel Blum (Reg. No. 26,710), Bartholomew Verdirame (Reg. No. 28,483), Maria C.H. Lin (Reg. No. 29,323), Joseph A. DeGirolamo (Reg. No. 28,595), Michael A. Nicodema (Reg. No. 33,199), Michael P. Dougherty (Reg. No. 32,730), Seth I. Atlas (Reg. No. 32,454), Andrew M. Riddles (Reg. No. 31,657), Bruce D. DeRenzi (Reg. No. 33,676), Michael M. Murray (Reg. No. 32,537) and Mark J. Abate (Reg. No. 32,527) of Morgan & Finnegan, L.L.P. whose address is: 345 Park Avenue, New York, New York, 10154; and Edward A. Pennington (Reg. No. 32,588) of Morgan & Finnegan, L.L.P., whose address is 1775 Eye Street, Suite 400, Washington, D.C. 20006.

[ ] I hereby authorize the U.S. attorneys and/or agents named hereinabove to accept and follow instructions from \_\_\_\_\_ as to any action to be taken in the U.S. Patent and Trademark Office regarding this application without direct communication between the U.S. attorneys and/or agents and me. In the event of a change in the person(s) from whom instructions may be taken I will so notify the U.S. attorneys and/or agents hereinabove.

1-00  
Full name of sole or first inventor Jukka JÄKÄRÄ  
Inventor's signature\* *Jukka Jäkärä* 2 August 2000  
date  
Residence Ahventie 1 B 22, FIN-65200 Vaasa, Finland FIX  
Citizenship Finnish  
Post Office Address Same as above.

004750-53550  
Full name of second joint inventor, if any Juha PATOLA  
Inventor's signature\* *Juha Patola* 02.08.2000  
date  
Residence Mäntymaantie 9 A 2, FIN-65200 Vaasa, Finland FIX  
Citizenship Finnish  
Post Office Address Same as above.

[X] ATTACHED IS ADDED PAGE TO COMBINED DECLARATION AND POWER OF ATTORNEY FOR SIGNATURE BY THIRD AND SUBSEQUENT INVENTORS FORM.

\* Before signing this declaration, each person signing must:

1. Review the declaration and verify the correctness of all information therein; and
2. Review the specification and the claims, including any amendments made to the claims.

After the declaration is signed, the specification and claims are not to be altered.

To the inventor(s):

The following are cited in or pertinent to the declaration attached to the accompanying application:

Title 37, Code of Federal Regulation, § 1.56

Duty to disclose information material to patentability.

(a) A patent by its very nature is affect with a public interest. The public interest is best served, and the most effective patent examination occurs when, at the time an application is being examined, the Office is aware of and evaluates the teachings of all information material to patentability. Each individual associated with the filing and prosecution of a patent application has a duty of candor and good faith in dealing with the Office, which includes a duty to disclose to the Office all information known to that individual to be material to patentability as defined in this section. The duty to disclose information exists with respect to each pending claim until the claim is canceled or withdrawn from consideration, or the application becomes abandoned. Information material to the patentability of a claim that is canceled or withdrawn from consideration need not be submitted if the information is not material to the patentability of any claim remaining under consideration in the application. There is no duty to submit information which is not material to the patentability of any existing claim. The duty to disclose all information known to be material to patentability is deemed to be satisfied if all information known to be material to patentability of any claim issued in patent was cited by the Office or submitted to the Office in the manner prescribed by §§1.97(b)-(d) and 1.98. However, no patent will be granted on an application in connection with which fraud on the Office was practiced or attempted or the duty of disclosure was violated through bad faith or intentional misconduct. The Office encourages applicants to carefully examine:

- (1) prior art cited in search reports of a foreign patent office in a counterpart application, and
- (2) the closest information over which individuals associated with the filing or prosecution of a patent application believe any pending claim patentably defines, to make sure that any material information contained therein is disclosed to the Office.

Title 35, U.S. Code § 101

Inventions patentable

Whoever invents or discovers any new and useful process, machine, manufacture, or composition of matter, or any new and useful improvement thereof, may obtain a patent therefor, subject to the conditions and requirements of this title.

Title 35 U.S. Code § 102

Conditions for patentability; novelty and loss of right to patent

A person shall be entitled to a patent unless -

- (a) the invention was known or used by others in this country, or patented or described in a printed publication in this or a foreign country, before the invention thereof by the applicant for patent,
- (b) the invention was patented or described in a printed publication in this or foreign country or in public use or on sale in this country, more than one year prior to the date of application for patent in the United States, or



(c) he has abandoned the invention, or

(d) the invention was first patented or caused to be patented, or was the subject of an inventor's certificate, by the applicant or his legal representatives or assigns in a foreign country prior to the date of the application for patent in this country on an application for patent or inventor's certificate filed more than twelve months before the filing of the application in the United States, or

(e) the invention was described in a patent granted on an application for patent by another filed in the United States before the invention thereof by the applicant for patent, or on an international application by another who has fulfilled the requirements of paragraphs (1), (2), and (4) of section 371(c) of this title before the invention thereof by the applicant for patent, or

(f) he did not himself invent the subject matter sought to be patented, or

(g) before the applicant's invention thereof the invention was made in this country by another had not abandoned, suppressed, or concealed it. In determining priority of invention there shall be considered not only the respective dates of conception and reduction to practice of the invention, but also the reasonable diligence of one who was first to conceive and last to reduce to practice, from a time prior to conception by the other ...

#### Title 35, U.S. Code § 103

##### Conditions for patentability; non-obvious subject matter

A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said matter pertains. Patentability shall not be negated by the manner in which the invention was made.

Subject matter developed by another person, which qualifies as prior art only under subsection (f) or (g) of section 102 of this title, shall not preclude patentability under this section where the subject matter and the claimed invention were, at the time the invention was made, owned by the same person or subject to an obligation of assignment to the same person.

#### Title 35, U.S. Code § 112 (in part)

##### Specification

The specification shall contain a written description of the invention, and of the manner and process of making and using it, in such full, clear, concise and exact terms also enable any person skilled in the art to which it pertains, or with which it is mostly nearly connected, to make and use the same, and shall set forth the best mode contemplated by the inventor of carrying out his invention.

#### Title 35, U.S. Code § 119

##### Benefit of earlier filing date in foreign country; right of priority

An application for patent for an invention filed in this country by any person who has, or whose legal representatives or assigns have, previously regularly filed an application for a patent for the same invention in a foreign country which affords similar privileges in the case of applications filed in the United States or to citizens of the United States, shall have the same effect as the same application would have if filed in this country on the date on which the application for patent for the same invention was first filed in such foreign country, if the application in

SEP. 2000 15:01 BERGREN 01 RE +306 9 635 3544 NO. 364 P. 0/11

this country is filed within twelve months from the earliest date on which such foreign application was filed; but no patent shall be granted on any application for patent for an invention which had been patented or described in a printed publication in any country more than one year before the date of the actual filing of the application in this country, or which had been in public use or on sale in this country more than one year prior to such filing.

Title 35, U.S. Code § 120

Benefit or earlier filing date in the United States

An application for patent for an invention disclosed in the manner provided by the first paragraph of section 112 of this title in an application previously filed in the United States, or as provided by section 363 of this title, which is filed by an inventor or inventors named in the previously filed application shall have the same effect, as to such invention, as though filed on the date of the prior application, if filed before the patenting or abandonment of or termination of proceedings on the first application or an application similarly entitled to the benefit of the filing date of the first application and if it contains or is amended to contain a specific reference to the earlier filed application.

Please read carefully before signing the Declaration attached to the accompanying Application.

If you have any questions, please contact Morgan & Finnegan, L.L.P.

FORM:COMB-DEC.NY

Rev. 5/21/98

ADDED PAGE TO COMBINED DECLARATION  
AND POWER OF ATTORNEY FOR  
SIGNATURE BY THIRD AND SUBSEQUENT INVENTOR

Full name of third joint inventor, if any Aarto PAREN

Inventor's signature\* Agustin Pae 280600 (28 June 2000)

Residence Kapteeninkatu 36 B 20, FIN-65200 Vaasa, Finland FIX date

Citizenship Finnish

Post Office Address Same as above.

Full name of fourth joint inventor, if any

**Inventor's signature\***

Residence \_\_\_\_\_ date \_\_\_\_\_

## Citizenship

**Post Office Address**

Full name of fifth joint inventor, if any

**Inventor's signature**

Residence \_\_\_\_\_ date \_\_\_\_\_

## Citizenship

**Post Office Address**

**Before signing this declaration, each person signing must:**

1. Review the declaration and verify the correctness of all information therein; and
2. Review the specification and the claims, including any amendments made to the claims.

**After the declaration is signed, the specification and claims are not to be altered.**